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FACTORS INFLUENCING ACCELERATED OZONE
TESTING AND WEATHERING OF CABLE COMPOSITIONS

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and P. B. Stickney

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INTRODUCTION

The Signal Equipment Supply Agency is desirous of developing a satisfactory short-term method for evaluating so-called "sunproofing" waxes for use in electrical insulation and jacketing compounds. At the present time these materials are evaluated by an outdoor exposure test of nine months' duration. It has been suggested in the literature that accelerated ozone-aging tests are less reliable than natural aging for this purpose. Natural aging has two basic drawbacks, of course. The first is the time required, and the second is the fact that such tests are obviously not reproducible.[1,2] A third, not so well recognized objection, could be raised. That is, there has been no straightforward demonstration that natural aging, under any conditions, correlates well with actual service life. Considering the number of variables that enter into natural aging and the time involved, there almost certainly are combinations of conditions that will correlate with service life - but even more certainly there are many more combinations that will not.

Oxygen, heat, light, and humidity are factors that are known to affect the aging of rubber.[3,4,5] However, it is also known that ozone is the factor of overriding importance to the type of aging for which sunproofing waxes are used. It seems reasonable, therefore, to use a test that

involves this factor, under accelerated but controlled conditions, rather than one involving an uncontrolled combination of a number of factors including incidental amounts of ozone.

Laboratory ozone exposure tests are being correlated with various outdoor exposures. One of the important accomplishments has been verification of the hypothesis that outdoor aging is quite unreproducible. It appears that atmospheric conditions at the time of initial exposure have a surprisingly important effect. The type of sample used, the rubber compound used, and the exact method of exposing the samples are also important.[6] Laboratory exposures have shown that waxes behave quite differently insofar as the variation of their effectiveness with temperature is concerned.[2] Control of temperature, control of ozone concentration, and probably use of at least two test temperatures are factors that will be required to develop a satisfactory accelerated ozone exposure test.

EXPERIMENTAL PROCEDURE

Compounding Cable Compositions

Three SBR[7] recipes were selected for study to determine whether the protective effectiveness of the waxes is different in different recipes. It has been reported[2] by other workers that waxes varied in their effectiveness, depending on the compound used. The recipes are designated V, N, and A. The recipes with the appropriate cure times and physical properties are shown in Table 1. Two of the recipes (V and N) are recommended for cable-jacketing use by suppliers of rubber chemicals, while the other is an ASTM test recipe. The compounds were not selected for their outstanding

ozone resistance and no added antioxidant or antiozonant was used in the formulation.

The waxes employed in this study are proprietary materials marketed by various suppliers as sunproofing waxes. The Signal Corps has examined and approved the use of a number of these in SBR wire and cable. A total of 17 different waxes were examined. Eleven of them have been approved by USASIMSA, and one was not approved. Of the remaining five, two have been reported to be satisfactory as sunproofing waxes but have not been submitted to USASIMSA for testing, and the other three are known to be nonprotective types. These three were included in the study to obtain comparative information.

In the preparation of SBR compositions for the study, the ingredients of each recipe, except the accelerators and wax, were mixed as a masterbatch in a Size "B" Banbury mixer to obtain a uniform base compound. Preliminary work showed that the presence of agglomerates of fillers at the surface results in a strained condition that is conducive to rapid ozone cracking[8,9,10]; therefore, good dispersion of fillers is essential in evaluating specimens for weather or ozone resistance. Accelerators were added to the masterbatched rubber on a 6 x 12-inch standard rubber mill. Also, the waxes, at a 4 parts per 100 parts of rubber level, were added as the last ingredient by mill mixing. MIL-I-3930B recommends a minimum of 4 parts by weight of sunproofing wax per 100 parts of rubber. The minimum wax content was chosen to obtain cracking faster for correlating accelerated and outdoor weathering data.

After compounding, the uncured sheets were stored for 24 hours at room temperature before vulcanization. Test sheets 6 x 6 x 0.075 inch were

prepared in an ASTM four-cavity chrome-plated mold between aluminum foil 0.001-inch thick. The aluminum foil was left on the cured sheets to avoid contamination. All test specimens were stored in the dark for at least 48 hours before exposure outdoors or in the ozone chamber.

Specimen Types for Exposure Study

Six types of test specimens were utilized to study the effect of specimen configuration on weathering. These are referred to as specimen types a, b, c, d, e, and f in the tables. Type a specimens were rectangular strips 1 inch wide by 6 inches long cut longitudinally from standard laboratory test sheets approximately 0.075-inch thick. Type b specimens were tapered strips 5-1/2 inches long by 1-1/2 inches wide at the wide end, and tapering down to 1/4-inch radius at the narrow end. The strips were also cut longitudinally from standard laboratory test sheets. Type a and b specimens were stretched to 20 per cent elongation. The elongation of the tapered strips was based on gage marks 5 inches apart on the unstretched specimen. Type c specimens were rectangular strips 1 inch wide by 3-3/4 inches long, cut longitudinally from standard laboratory test sheets approximately 0.075-inch thick, and bent into loops. Type d specimens were triangular rods 3/4-inch wide at the base by 1/2-inch high by 10 inches long, and bent into a loop around a mandrel. Type e specimens were simulated cable 5/16-inch in diameter by 30 inches long, wrapped around a mandrel in spiral loops. Type f specimens were Die D dumbbells stretched initially to 20 per cent and maintained under constant load.

The first three types of specimens, a, b, and c, were prepared from test sheets according to ASTM D518-57T, Methods A, B, and C. The fourth, d,

was prepared according to ASTM D1171-59. The fifth specimen type, e, was molded in a specially designed mold to simulate No. 18 AWG two-conductor cable, Underwriters Laboratories Type SJ. The sixth specimen type, f, was cut from a molded sheet to conform with ASTM D412-51T Die D dumbbell. The Die D dumbbell specimens were the only ones used for indoor accelerated ozone tests.

Sample Preparation for Outdoor Weathering

For outdoor static exposure, duplicate samples were mounted on lacquered wooden racks and aluminum mandrels and fastened according to ASTM D518-57T, Methods A, B, and C, and ASTM D1171-59. A strain of 20 per cent was selected as this strain has been considered to be the most critical strain for rapid cracking[8]. A special test rack was built to hold the dumbbell specimens extended by means of constant load. The cable samples were wrapped around a lacquered aluminum mandrel having a diameter four times the outside diameter of the cable to give a strain approximating 20 per cent. A minimum of five turns of cable were wrapped around the mandrel. After mounting, all specimens were cleaned of surface wax and allowed to rest for a minimum of 48 hours in a darkened area at room temperature before being exposed in accordance with ASTM D1149-57T. The mounted samples were placed on outdoor racks at an angle of 45 degrees from the vertical. All outdoor racks faced south. The dumbbell specimens were exposed hanging vertically.

The outdoor weathering was conducted at one location: roof of Building 7, Battelle Memorial Institute, Columbus, Ohio, at about 40° north latitude. The location represents yearly a variable climate, i.e., hot dry summer and moderately cold winters. Climatic conditions for the test period

are summarized in Table 3. The ozone concentration for the area is not known and has not been measured, but it is believed to be only moderate.

Ten sets of SBR test specimens have been exposed to outdoor weathering. The last six sets exposed were dumbbell specimens stretched under constant load. The months of initial exposure of the different sets of specimens for outdoor weathering are shown in Table 2. Observations for cracking were made at frequent intervals during the early stages of outdoor exposure to detect the earliest appearance of cracks. The samples were examined with a 7X magnifying glass according to ASTM D1149-T to show when the first crack was formed. In this study, the immediate interest was the determination of initial cracking. The depth, width, and number of cracks were not taken into consideration.

Sample Preparation for Accelerated Ozone Exposure

For correlating data with long-term outdoor exposure, samples were prepared from Recipes N and V for accelerated testing in a Mast ozone test chamber, Model 700-1. Dumbbell specimens only were tested in the ozone chamber. Lead weights (lead pellets in bottles) were clamped to the dumbbells to produce an initial elongation of 20 per cent. The load remained constant throughout the test period. A protective enamel film was applied around the shoulder of the dumbbells to prevent premature breaks at this point of stress concentration.

The mounted specimens free of surface wax were allowed to condition for a minimum period of 48 hours at room temperature. Exposures were made at temperatures that ranged [illegible] 1 F to 120 F at concentrations of 50 and 100 pphm of ozone for differ[illegible] periods of time. The times of exposure varied

from 1 to 168 hours (7 days), depending on the nature of the test. Cracking was observed through the telescope of a cathetometer aimed at the samples through a glass window of the ozone chamber. As was mentioned previously, the first detectable crack in a specimen was recorded as a failure.

DISCUSSION OF RESULTS

The objective of this study was to develop a short-term method of testing the protective efficiency of sunproofing waxes in SBR wire and cable compounds. For this study, specimens were exposed outdoors and in the ozone chamber in an attempt to obtain correlative data that would lead to the development of an accelerated test. Ten sets of SBR test specimens were exposed to outdoor weathering with a minimum span of 1 month between sets.

The first set was exposed during August, 1960, and the tenth set during May, 1961. Tables 2 and 3 present the months of initial exposure of the various sets, and the range of climatic conditions for each month. A summary of the results of the exposures is shown in Table 4. The time required for individual specimens to crack is shown in Tables 5, 6, and 7. The results obtained from accelerated ozone studies made in the Mast ozone test chamber are shown in Tables 8 and 9.

Outdoor Weathering

The first set of test specimens was prepared and exposed in August, 1960. During this month, the temperatures varied between an average daily low of 64 and average daily high of 85 F. Data in Table 4 show that more of the stretched rectangular, a, samples for all three SBR recipes cracked than did stretched-tapered, b, the bent-loop, c, and mandrel-wrapped triangular

specimens, d. For this type of specimen most of the cracking occurred during the first month of outdoor exposure, as shown in Tables 5, 6, and 7; after 10 months of exposure, all of the 13 waxes studied failed to protect Recipe V. The large number of failures for the rectangular specimens is probably due to the method of exposure. For simplicity of preparation, the first set of rectangular specimens was mounted on a solid block of wood. There was no circulation of air around the samples, and they retained higher temperatures longer since they were in immediate contact with the block of wood. Less cracking was noted with the other types of specimens in the following descending order: the triangular, tapered, and bent loop. Only two of the bent-loop specimens cracked. These samples were prepared from Recipe A and contained Waxes 2 and 10. It was further observed that most of the specimens exposed initially during August, 1960, cracked within the first 2 months. Most of the remaining specimens did not crack even after 10 months of exposure.

Sets 2 and 3 were exposed during October and November, 1960, with average low and high temperatures from approximately 35 to 70 F. After 7 to 8 months of exposure, specimens containing Waxes 2, 7, and 10 were the only ones that cracked (one bent-loop sample containing Wax 7 cracked for Set 3). All three of the different specimen types were affected. For Sets 2 and 3, Recipe V had the least number of specimens crack; Recipe A had the greatest number.

Set 4 was exposed during January, 1961, with average low and high temperatures from 15 to about 32 F. In this set, more samples had cracked in the first 5 months than for Sets 2 and 3. Samples containing Waxes 2 and 10 were among the first to crack as was generally true with Sets 2 and 3.

Set 4 was exposed during the coldest weather experienced in the test period. The observations noted for specimens of this set correlated with accelerated ozone data at the lower temperatures of exposure.

Sets 5 to 10 consisted of ASTM Die D dumbbells initially stretched 20 per cent under constant load. In these sets, specimens containing Waxes 2 and 10 were among the first to crack. These results are in general agreement with those obtained with Sets 2, 3, and 4, using different types of specimens. In comparing Sets 4 and 5, it appears that for outdoor exposure in cold weather the mandrel-wrapped simulated wire is the most rigorous test, with the constant-load dumbbell nearly as severe. It must be borne in mind, however, that the simulated wire can readily be overstressed in mounting; therefore, the results may not be strictly comparable with actual wire results. It is also obvious that Waxes 2 and 10 are less effective than most of the others in protecting against cracking in cold service.

In summarizing the results of outdoor exposure, it was observed that:

(1) The protective effectiveness of sunproofing waxes varied with the temperature at the time of initial exposure. Certain waxes were more efficient protective agents in hot weather than in cold weather.

(2) The rate of cracking differed for specimens of different geometry and applied stress. In general, dumbbell specimens with constant load cracked more rapidly than did other types of specimens, with the possible exception of mandrel-wrapped simulated wire.

(3) The cracking varies with the type of recipe. The waxes were more efficient in Recipe V than in N or A.

Accelerated Ozone Studies

A laboratory ozone test chamber was used for accelerated aging tests. In using this type of equipment, there are a number of precautions that must be observed in order to minimize variability of operating conditions. The ozone concentration within the chamber, for example, is dependent on the contents of the chamber, i.e., number and type of racks used for specimen mounting, number and size of specimens. Generally speaking, aluminum racks and glass containers had a minimum effect on the ozone concentration. Opening the cabinet door to examine specimens introduces problems both as regards ozone concentration and temperature. For this reason, the specimens were routinely examined with the telescope of a cathetometer, through the glass door of the cabinet.

Ozone concentration in the cabinet is also somewhat dependent on ambient atmospheric conditions, notably humidity. Therefore, regular checks and adjustments must be made on the concentration.

A further problem involved in improving the reproducibility of the test is uniform mounting of the specimens. It is very difficult in routine tests to get uniform strain on the specimens without stress concentration. Since ozone cracking is greatly dependent on the amount of applied strain, this is a serious problem. Small surface imperfections and points of stress concentration, e.g., at the shoulders of the specimens, can be covered with a protective film to prevent premature cracking.

Type D dumbbells were chosen for this study because they can be cut conveniently from actual cable insulation. Their small size also makes it

relatively simple to maintain uniform ozone concentration with a number of specimens. Constant load was applied to the specimens rather than constant elongation. It had been established that this constituted a rigorous type of exposure for outdoor testing and it was used in the laboratory in order to accelerate the test.

Effect of Temperature

Temperature is one of the most important variables affecting the ozone cracking of SBR containing sunproofing waxes. Tables 8 and 9 show that, at 120 F and 50 pphm of ozone, all of the samples of Recipes N and V containing 15 different waxes cracked within 1 to 2 hours, except those containing Waxes 2, 8, and 10. With Recipe N, specimens containing Waxes 2, 8, and 10 did not crack even after 50 test hours. However, with Recipe V, and the same three waxes, cracking was observed in less than 24 hours, and results were not as reproducible as for Compound N.

At 100 F and 50 pphm of ozone, the specimens generally took longer to crack. For Compound N, most of the specimens did not crack after 100 hours of testing. Here again, it was noticed that for both Compounds N and V, specimens containing Waxes 2, 8, and 10 took the longest time to crack. There was fairly good reproducibility between several sets of exposed samples.

At test temperatures of 60 to 67 F, and for 94 to 195 hours' exposure, specimens containing Waxes 2, 4, 7, 10, and 14 in Compound N cracked in a shorter time than the others, with those containing Waxes 2 and 10 cracking first. No specimens cracked for Recipe V after 168 hours of testing.

At a test temperature ranging from 1 to 42 F, samples of Recipe N containing the waxes cracked more readily than at test temperatures of 60 F. Here again, the first specimens to crack were those containing Waxes 2 and 10. In Compound V, only specimens with Waxes 2, 4, 8, 9, and 10 cracked. It appears that the protective efficiency of some waxes begins to change at about 60 F. It was pointed out earlier that, in outdoor exposure, Waxes 2 and 10 lost their efficiency in the colder weather. While Compound V was generally superior to Compound N in outdoor exposures, N was superior in high-temperature laboratory exposures.

Ozone Concentration

Limited studies were made with exposures to ozone concentrations greater than 50 pphm. Table 9 shows results of tests at 100 pphm of ozone and temperatures of 60 to 67 F. Some additional specimens cracked at the higher concentrations and the time to cracking was lower for those that cracked at both concentrations.

Compounding Variables

The data obtained show that the type of compound has a definite effect on the rate of ozone cracking. (See also reference 11.) Recipes N, A, and V, having different fillers, softeners, curing systems, and antioxidants, behaved differently when they were exposed to outdoor weathering and in the ozone chamber. The state of cure is considered to be an important factor in obtaining maximum ozone resistance.[11] However, the state of cure and its effect on compositions exposed to accelerated and natural weathering has not been investigated in the present study.

Comparison of Outdoor Weathering With Accelerated Ozone Aging

The data in Tables 5 through 9 show that there is a correlation between similar specimens exposed to accelerated ozone and to long-term natural weathering. However, additional data from outdoor weathering is necessary for better comparisons with specimens exposed in the ozone chamber. It would be particularly desirable to carry out outdoor aging of actual wire samples.

In outdoor exposure, the atmospheric conditions, such as sunlight, humidity, temperature, rain, snow, ozone concentration, etc., vary widely from time to time, and would be expected to give a greater variation in data than the ozone chamber in which reasonably constant conditions can be maintained throughout the test period.[9,10] However, it has been possible to correlate the data obtained from the ozone-chamber studies with trends in outdoor weathering resulting principally from temperature changes and the type of recipe used. Therefore, additional information on some of the other conditions should give data for even better correlation.

SUMMARY AND CONCLUSIONS

The ozone resistance of a number of SBR specimens containing different sunproofing waxes ~~has been~~ studied under various conditions in the ozone chamber and in outdoor weathering. Information obtained from the study showed that:

(1) In outdoor exposure, the temperature at the time of initial exposure has a decided effect on the rate of cracking of SBR compositions containing different waxes. Specimens with

certain waxes tended to crack sooner when they were first exposed to outdoor weathering in the summer (high temperatures) and winter (low temperatures) than when exposed in the fall (medium temperatures). These observations correlated with the data obtained in the ozone chamber at 100, 60, and 20-40 F.

(2) Specimens containing certain waxes that did not crack at the higher temperatures cracked quickly when exposed at low temperatures. This behavior indicates that some waxes have a greater protective efficiency at the higher temperature than at the lower temperature, while for others, the reverse is true.

(3) The geometry of the specimen and the manner of exposure appear to affect the rate of cracking. After 10 months of outdoor exposure, most of the bent-loop specimens and tapered specimens stretched to 20 per cent constant elongation did not crack or cracked after 6 and 5 months of exposure, respectively. Most of the stretched rectangular strip and triangular rod specimens cracked after 1 and 2 months, respectively, for similar compositions and exposure.

(4) The type of recipe employed has a definite influence on stability when exposed to weathering and ozone. The solubility of the wax and its rate of diffusion to the surface to form a protective film appear to be dependent upon the ingredients in the compound and the state of cure. Of the three recipes employed in this study, one showed exceptional re-

sistance to outdoor weathering, one moderate resistance, and the other poor resistance

On the basis of these results, satisfactory correlation with outdoor exposure may only be obtained by evaluating ozone test data at at least two temperatures, e.g., 40 and 100 F.

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TABLE 1. SBR RECIPES FOR WIRE- AND CABLE-JACKETING COMPOUNDS

	Recipe Type (Parts Per Hundred of Rubber)		
	(V)	(N)	(A)
SBR 1503	100	100	100
Zinc Oxide	5	5	5
Stearic Acid	1.5	2	1.5
Reogen	5	--	--
Cumar MH2-1/2	--	20	--
Agerite Resin D	2	--	--
Agerite White	0.5	--	--
Flexamine	--	1	--
BLE	--	--	1
Hard Hydrocarbon	30	--	--
Suprex Clay	--	30	--
SAF Black	40	--	--
P-33 Black	40	20	--
FEF Black	--	40	50
Altax	1	--	0.8
Bismate	1	--	--
Beutene	--	1	--
Monex	--	0.6	--
TMTDS	--	--	0.15
Sulfur	2.25	1.85	1.75
Wax	4 (a)	4 (a)	4 (a)
	Physical Properties		
Cure Time, min	15	15	30
Temperature, F	320	320	302
Tensile Strength, psi	2200	2410	2190
Modulus at 300%, psi	1780	1900	1590
Elongation, %	370	450	440
Hardness, Shore A2	72	72	64

(a) Wax concentration four parts per hundred parts of rubber. Minimum amount specified by MIL-I-3930B (August 29, 1960).

TABLE 2. MONTH OF INITIAL EXPOSURE OF DIFFERENT SETS OF SPECIMEN TYPES FOR LONG-TERM OUTDOOR WEATHERING

Set	Specimen Type (a)					
	a	b	c	d	e	f
1	August, 1960	August, 1960	August, 1960	August, 1960	--	--
2	October, 1960	October, 1960	November, 1960	--	--	--
3	November, 1960	November, 1960	November, 1960	--	--	--
4	January, 1961	January, 1961	January, 1961	January, 1961	January, 1961	--
5	--	--	--	--	--	January, 1961
6	--	--	--	--	--	February, 1961
7	--	--	--	--	--	March, 1961 (b)
8	--	--	--	--	--	March, 1961
9	--	--	--	--	--	April, 1961
10	--	--	--	--	--	May, 1961 (c)

(a) Refer to text for type of test specimen.
(b) This set of specimens prepared from 0.040-inch-thick test sheets.
(c) This set contained specimens from V and N recipes only.

TABLE 3. CLIMATOLOGICAL DATA, COLUMBUS, OHIO[a], AUGUST, 1960, THROUGH MAY, 1961

Month	Year	Temperature, F[b] High	Temperature, F[b] Low	Sunshine Hours	Sunshine %	Total Precipitation, inches Water	Total Precipitation, inches Snow
August	1960	85.4	64.3	330	78	2.48	0
September	"	80.1	56.4	231	62	0.83	0
October	"	66.8	43.3	194	56	2.55	0
November	"	54.1	34.6	109	37	1.60	0.8
December	"	34.3	15.9	102	35	1.62	17.3
January	1961	31.7	15.2	164	55	0.65	7.6
February	"	45.1	28.6	129	43	2.9	13.1
March	"	53.6	37.0	186	50	4.83	0.7
April	"	55.3	38.8	161	41	4.58	2.7
May	"	68.4	44.6	304	68	2.9	0

(a) Columbus, Ohio, Municipal Airport.

(b) Average daily high and average daily low temperatures.

TABLE 4. SUMMARY DATA OF OUTDOOR EXPOSURE OF TEN SETS OF SBR COMPOUNDS CONTAINING 17 DIFFERENT WAXES

(Numbers refer to type of wax used in specimens that failed.)

Specimen Type	Recipe Type V	N	A
	Set 1[a]. After 10 Months of Exposure (Exposure Began August 13-25, 1960)		
a	All specimens cracked except 2	All specimens cracked	All specimens cracked
b	--	9 and 13[b]	2, 9[b], and 10
c	--	--	2 and 10
d	--	1[b], 4[b], 5[b], and 10[b]	All specimens cracked
	Set 2[a]. After 7-8 Months of Exposure (Exposure Began October 23-November 11, 1960)		
a	--	10	2 and 10
b	--	2[b] and 10	2 and 10
c	2 and 10	2[b]	10[b]
	Set 3[c]. After 7 Months of Exposure (Exposure Began November 16-20, 1960)		
a	--	--	2 and 10
b	--	2	2 and 10
c	--	2 and 7[b]	2 and 10
	Set 4[d]. After 5 Months of Exposure (Exposure Began January 23, 1961)		
a	15, 16, and 17	2, 15, 16, and 17	2, 10, 15, 16, and 17
b	15, 16, and 17	2, 4, 10, 15, 16, and 17	2, 3, 4, 8[b], 9[b], 10, 14[b], 15, 16, and 17
c	15, 16, and 17	1[b], 2, 5[b], 9, 10, 15, 16, and 17	2, 9, 10, 15, 16[b], and 17
d	16 and 17	2, 7[b], 10, 12[b], 15, 16, and 17	2, 3[b], 10, 15, 16, and 17
e	2[b], 10[b], 14[b], 15, 16 and 17	1[b], 2, 4, 7[b], 9, 10, 11, 14[b], 15, 16, and 17	2, 3, 4, 7[b], 8, 10, 14[b], 15, 16, and 17
	Set 5[d]. After 5 Months of Exposure (Exposure Began January 16, 1961)		
f	16 and 17	2, 3, 4, 10, 14, 16, and 17	2, 3, 4, 7, 10, 14, and 17
	Set 6[d]. After 4 Months of Exposure (Exposure Began February 6, 1961)		
f	17	2, 10, 16, and 17	2, 10, 16, and 17
	Set 7[d,f]. After 3 Months of Exposure (Exposure Began March 6, 1961)		
f	10, 16, and 17	2, 10, 16, and 17	2, 8, 10, 16, and 17
	Set 8[d]. After 3 Months of Exposure (Exposure Began March 13, 1961)		
f	17	3, 10, 16, and 17	2, 10, 16, and 17

TABLE 4. (Continued)

Specimen Type	Recipe Type		
	V	N	A
	Set 9[(e)]. After 2 Months of Exposure (Exposure Began April 18, 1961)		
f	--	15	15
	Set 10[(e)]. After 1 Month of Exposure (Exposure Began May 15, 1961)		
f	--	--	None exposed

Notes: Controls containing no wax cracked in 2-7 days.
Where no numbers are listed, specimens have not cracked.

(a) Set contained 13 different waxes.
(b) One specimen of two exposed has cracked.
(c) Set contained 14 different waxes.
(d) Set contained 17 different waxes.
(e) Set contained 15 different waxes.
(f) Specimens prepared from 0.040-inch-thick test sheet.

TABLE 5. RESULTS OF LONG-TERM OUTDOOR WEATHERING OF TEN SETS OF SPECIMENS OF RECIPE V

	Time to Crack, months																				
Set No.:	1				2			3			4					5	6	7	8	9	10
Time of Exposure, months:	10				7-8			7			5					5	4	3	3	2	1
Date of Initial Exposure:	August 13-25, 1960				October 23-November 11, 1960			November 16-20, 1960			January 23, 1961					1/16/61	2/6/61	3/6/61	3/13/61	4/18/61	5/15/61
Specimen Type(a):	a	b	c	d	a	b	c	a	b	c	a	b	c	d	e	f	f	f	f	f	f
Wax No.																					
1	2	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
2	X	X	X	X	X	X	1	X	X	X	X	X	X	X	<3(b)	X	X	<2	X	X	X
3	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
4	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
5	10	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	<5	X	X	X	X
6	1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	<5	X	X	X	X
7	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
8	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
9	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
10	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	5(b)	X	X	<2	X	X	X
11	2	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
12	9-10	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
13	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
14	--	--	--	--	--	--	--	--	X	X	X	X	X	X	5(b)	X	X	X	X	X	X
15	--	--	--	--	--	--	--	--	--	--	<3	5	<4	<5	5	X	<5	3	X	X	X
16	--	--	--	--	--	--	--	--	--	--	<4	<1	<2	<2	3	<4	<4	<1	X	--	--
17	--	--	--	--	--	--	--	--	--	--	5	<1	<2	<2	<2	2	<2	<1	<1	--	--

Notes: X indicates specimens have no cracks visible to magnification of 7X.

All specimen types were exposed in duplicate except Type f which was exposed singly.

Specimen Types (a) and (b) stretched to 20 per cent elongation. Specimen Type (c) was bent into a loop. Specimen Types (d) and (e) bent into loops around a mandrel. Specimen Type (f) stretched to 20 per cent under constant load.

All test specimens were prepared from 0.075-inch-thick test sheets except Set 7 specimens which were prepared from 0.040-inch-thick test sheet.

(a) Refer to text for type of test specimen.
(b) One of two specimens exposed cracked.

TABLE 6. RESULTS OF LONG-TERM OUTDOOR WEATHERING OF TEN SETS OF SPECIMENS OF RECIPE N

	Time to Crack, months																				
Set No.:	1				2			3			4					5	6	7	8	9	10
Time of Exposure, months:	10				7-8			7			5					5	4	3	3	2	1
Date of Initial Exposure:	August 13-25, 1960				October 23 - November 11, 1960			November 16-20, 1960			January 23, 1961					1/16/61	2/6/61	3/6/61	3/13/61	4/18/61	5/15/61
Specimen Type(a):	a	b	c	d	a	b	c	a	b	c	a	b	c	d	e	f	f	f	f	f	f
Wax No.																					
1	<1	X	X	10(b)	X	X	X	X	X	X	X	X	<1(b)	X	5	X	X	X	X	X	X
2	4	X	X	X	X	3(b)	1(b)	X	2	1	1	1	<1	<1	<1	<1	<1	<1	<1	X	X
3	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	2	X	X	X	X	X
4	<1	X	X	2(b)	X	X	X	X	X	X	X	1	X	X	<4	<1	X	X	X	X	X
5	<1	X	X	2(b)	X	X	X	X	X	X	X	X	6	X	X	X	X	X	X	X	X
6	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
7	<1	X	X	X	X	X	X	X	X	<4	X	X	X	<5	5	X	X	X	X	X	X
8	1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
9	<1	<1	X	X	X	X	X	X	X	X	X	X	6	X	5	X	X	X	X	X	X
10	<1	X	X	2(b)	6-7	4	X	X	X	X	X	2	<1	<1	<1	<1	<3	<1	<2	X	X
11	1	X	X	X	X	X	X	X	X	X	X	X	X	X	5	X	X	X	X	X	X
12	9-10	X	X	X	X	X	X	X	X	X	X	X	X	<4(b)	X	X	X	X	X	X	X
13	<1	10	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
14	--	--	--	--	X	X	X	X	X	X	X	X	X	X	5	<1	X	X	X	X	X
15	--	--	--	--	--	--	--	--	--	--	<4	<4	4	<4	4	X	4	X	X	<2	X
16	--	--	--	--	--	--	--	--	--	--	8(c)	8(c)	8(c)	8(c)	1	7(c)	2(c)	8(c)	7(c)	--	--
17	--	--	--	--	--	--	--	--	--	--	8(c)	8(c)	8(c)	8(c)	1	7(c)	2(c)	8(c)	15(c)	--	--

Notes: X indicates specimens have no cracks visible to magnification of 7X.
All specimen types were exposed in duplicate except Type (f) which was exposed singly.
Specimen Types (a) and (b) stretched to 20 per cent elongation. Specimen Type (c) was bent into a loop.
Specimen Types (d) and (e) bent into loops around a mandrel. Specimen Type (f) stretched to 20 per cent under constant load.
All test specimens were prepared from 0.075-inch-thick test sheet except Set 7 specimens which were prepared from 0.040-inch-thick test sheet.

(a) Refer to text for type of test specimen.
(b) One of two specimens exposed cracked.
(c) Days to crack.

TABLE 7. RESULTS OF LONG-TERM OUTDOOR WEATHERING OF NINE SETS OF SPECIMENS OF RECIPE A

	Time to Crack, months																			
Set No.:	1				2			3			4					5	6	7	8	9
Time of Exposure, months:	10				7-8			7			5					5	4	3	3	2
Date of Initial Exposure:	August 13-25, 1960				October 23-November 11, 1960			November 16-20, 1960			January 23, 1961					1/16/61	2/6/61	3/6/61	3/13/61	4/18/61
Specimen Type(a):	a	b	c	d	a	b	c	a	b	c	a	b	c	d	e	f	f	f	f	f
Wax No.																				
1	9-10	X	X	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
2	9-10	5	9	3	<2	1	X	1	<2	1	<1	<1	<1	<1	<1	<1	<1	<1	<1	X
3	<1	X	X	1	X	X	X	X	X	X	X	1	X	<4	<1	<1	X	<2	X	X
4	<1	X	X	<1	X	X	X	X	X	X	X	1	X	X	<1	<1	X	X	X	X
5	<1	X	X	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
6	9-10	X	X	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
7	<1	X	X	<9	X	X	X	X	X	X	X	X	X	X	5	<1	X	<2	X	X
8	<1	X	X	<1	X	X	X	X	X	X	X	1	X	X	1	X	X	<2	X	X
9	<1	5(b)	X	<1	X	X	X	X	X	X	X	<2	6	X	<1	X	<4	X	X	X
10	<1	5	6	5	<2	<1	6(b)	1	<2	1	<1	<1	<1	<1	<1	<1	<1	<1	<1	X
11	1	X	X	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
12	1	X	X	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
13	<1	X	X	<1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
14	--	--	--	--	--	--	--	X	X	X	X	1	X	X	3	<1	X	X	X	X
15	--	--	--	--	--	--	--	--	--	--	5	<5	4	<5	2	X	4	3	X	<2
16	--	--	--	--	--	--	--	--	--	--	1	1	8(c)	1	2	X	1	<1	<1	--
17	--	--	--	--	--	--	--	--	--	--	1	1	8(c)	2	5	2	1	<1	<1	--

Notes: X indicates specimens have no cracks visible to magnification of 7X.

All specimen types were exposed in duplicate except Type (f) which was exposed singly.

Specimen Types (a) and (b) stretched to 20 per cent elongation. Specimen Type (c) was bent into a loop. Specimen Types (d) and (e) bent into loops around a mandrel. Specimen Type (f) stretched to 20 per cent under constant load.

All test specimens were prepared from 0.075-inch-thick test sheet except Set 7 specimens which were prepared from 0.040-inch-thick test sheet.

(a) Refer to text for type of test specimen.
(b) One of two specimens exposed cracked.
(c) Days to crack.

TABLE 8. RESULTS OF ACCELERATED OZONE STUDY OF SBR SPECIMENS OF RECIPE V

	Time to Crack, hours – Specimen Type f														
Conditioning Time, hours:	65	88	45	71	71	85	68	47	66	66	143	261	50	48	185
Time of Test, hours:	24	24	24	56	56	168	70	100	192	192	168	168	30	168	168
Ozone, pphm:	50	50	50	50	50	50	50	50	50	50	50	50	50	50	50
Chamber Temperature, F:	120	120	120	120	120	100	100	100	100	100	60	60	56-64	20-42	1-30
Set No:	1	2	3	4	5	1	2	3	4	5	1	2	3	1	2
Wax No.															
1	1-3/4	2	5	3	2	X	25-1/2	8-24	4	5	X	X	X	X	X
2	4	4	6	X	X	X	26	8-24	25	26	X	X	X	84	130
3	1	1	2	3	3-1/4	X	5-20	8-24	4	3-1/2	X	X	X	X	X
4	1	1-1/2	1-3/4	3	3-1/4	X	5-20	8-24	4-1/2	4-1/2	X	X	X	X	36
5	1-1/2	1-1/4	1-1/2	3	3	4	5	5	3	3-1/2	X	X	X	X	X
6	1-1/2	1-3/4	1-3/4	3	2-1/4	4	5-20	8-24	3-1/2	5	X	X	X	X	X
7	1-1/2	1-1/2	2	X	5-1/2	X	25-1/2	X	6-21	6-21	X	X	X	X	X
8	1-1/2	8-24	1-3/4	X	X	X	22-1/2	X	4	4	X	X	X	X	148
9	1-1/2	1-1/4	1-3/4	3	1-1/4	X	22-1/2	X	4	5-1/2	X	X	X	X	160
10	X	8	X	X	X	2	X	X	X	X	X	X	X	48	X
11	1	1-1/2	1-3/4	3	2	2	2	3-1/2	4	3-1/2	X	X	X	X	X
12	3/4	1-1/2	1-3/4	3	2-3/4	3-18	1-1/2	6-1/2	4-1/2	3-1/2	X	X	X	X	X
13	3/4	1-1/2	1-3/4	3	2-3/4	X	1-1/2	98-1/2	4	2-1/2	X	X	X	X	X
14	1-1/4	3	1-3/4	3	2-3/4	2	5-20	X	5-1/2	5-1/2	X	X	X	X	X
15	--	--	--	1-1/2	1-1/4	2	2	2	4	3-1/4	X	X	X	X	X

Notes: X indicates specimens had no cracks visible to cathetometer.
One specimen exposed per test.
Specimens prepared from 0.075-inch-thick test sheet.
Specimens stretched to 20 per cent under constant load.

TABLE 9. RESULTS OF ACCELERATED OZONE STUDY OF SBR SPECIMENS OF RECIPE N

	Time to Crack, hours - Specimen Type f																			
Conditioning Time, hours:	117	41	42	71	71	85	68	47	69	69	143	261	66	66	43	91	46	91	48	185
Time of Test, hours:	120	96	72	168	168	168	70	100	350	350	168	168	195	195	115	94	115	94	168	168
Ozone, pphm:	50	50	50	50	50	50	50	50	50	50	50	50	50	50	100	100	100	100	50	50
Chamber Temperature, F:	120	120	120	120	120	100	100	100	100	100	60	60	60	60	60-67	60-64	60-67	60-64	20-42	1-30
Set No.	1	2	3	4	5	1	2	3	4	5	1	2	1(a, b)	2(a, c)	1	2	3(a)	4(a)	1	2
Wax No.																				
1	1	1	1-1/2	1-1/2	1-1/4	X	X	3-1/2	5-21	5-21	X	X	X	X	X	3-1/2	X	X	3	60
2	120	96	72	X	56	3-18	X	X	X	X	167	X	X	22-1/2	5-1/4	2-1/2	1-3/4	30-94	11	47
3	1	1-1/2	1-1/2	1-1/2	1	X	X	X	X	5-21	X	X	X	X	X	X	X	X	150	108
4	1	3/4	1	1-1/2	3/4	X	X	X	1	X	X	X	X	X	8-1/2	2-1/2	96	30-94	X	116
5	1-1/2	1-3/4	1-1/4	1-1/2	3/4	2	25-1/2	8-24	1-1/2	1	X	X	X	X	X	X	X	X	X	X
6	1	1	1-1/4	1-1/2	3/4	1	1	2	1-1/2	1	X	X	X	X	X	X	X	X	X	155
7	1	1-1/4	1-1/2	56	56	X	X	X	X	X	X	X	X	29-1/2	X	3-1/2	X	X	12	108
8	1	96	72	X	56	X	X	X	X	X	X	X	X	X	X	X	X	X	150	47
9	1	1	1	1-1/2	1	X	X	X	X	X	X	X	X	X	X	X	X	X	150	47
10	X	96	X	X	X	X	X	X	X	X	45	30-45	X	22-1/2	X	3-1/2	X	X	12	37
11	1	1-1/4	1-1/4	1-1/2	3/4	X	1	X	2-1/2	1-1/2	X	X	X	X	X	X	X	X	X	X
12	1	1	1	2	3/4	3-18	2	33-97	3-1/2	1-1/2	X	X	X	X	X	X	X	X	150	X
13	1	1	1	1-1/2	3/4	X	5-20	X	3-1/2	4	X	X	X	X	X	X	X	X	45	X
14	1	2	1-1/2	X	2-3/4	X	X	X	X	X	X	X	X	X	13	30-94	X	X	150	118
15	--	--	--	1-1/2	3/4	2	1	2	1-1/2	1	X	X	X	X	64-88	X	X	X	X	X

Notes: X indicates specimens had no cracks visible to cathetometer.
One specimen exposed per test.
Specimens prepared from 0.075-inch-thick test sheet.
Specimens stretched to 20 per cent under constant load.

(a) Specimens extended to 20 per cent constant elongation.
(b) Specimens oven aged 40 hours at 100 C before exposure to ozone.
(c) Specimens aged 48 hours at room temperature before exposure to ozone.